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Towards energy efficiency through an optimized use of wood: the development of natural hydrophobic coatings that retain moisture-buffering ability

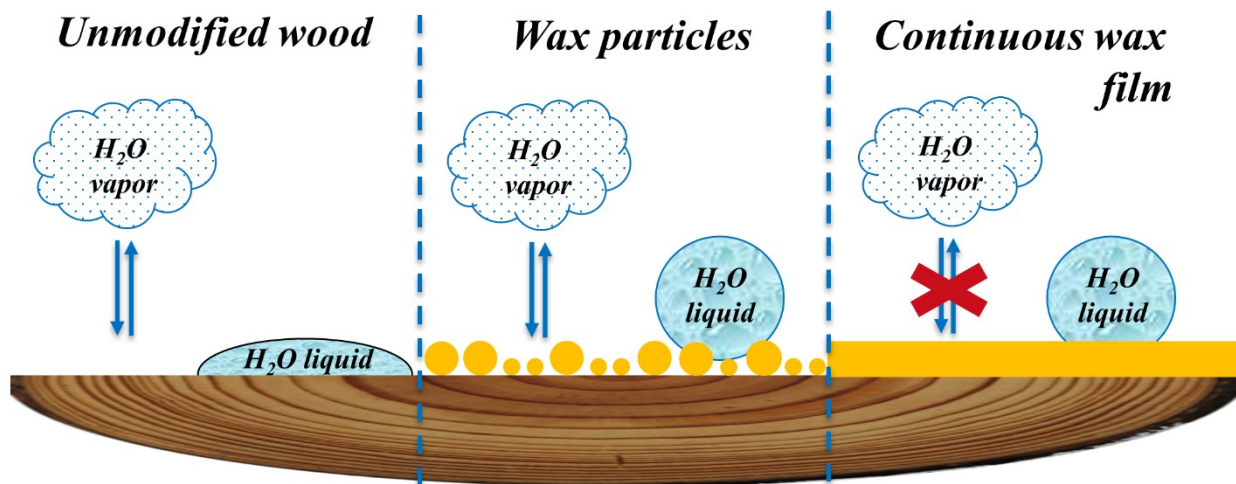
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Graphical abstract



Highlights

- We propose a new surface treatment for wood based on natural wax particles.
- The developed coating was compared to continuous wax film, lacquer and linseed oil.
- The treatment increases the hydrophobicity of the wooden surface.
- The ability of the surface to buffer moisture vapor was enhanced by the coating.

16 **Abstract**

17 The hygroscopicity of a wooden material or the ability to absorb, store and release moisture helps
18 to naturally regulate the indoor climate by dampening humidity variations and avoiding extremes.
19 This phenomenon, known as moisture buffering, is an energy-efficient way of moderating
20 moisture levels in a living space, improving air quality, and influencing the health and comfort of
21 the occupants. This work focused on developing a surface treatment that preserves the natural
22 ability of timber to buffer moisture vapor whilst increasing the resistance to liquid water. For this
23 purpose we suggest a method based on a natural non-continuous coating of hydrophobic Carnauba
24 wax particles. The coating was compared, in terms of water repellency and moisture buffering
25 efficiency, to a continuous wax film and conventional coating methods like lacquer and linseed
26 oil. The resistance of the surfaces to liquid water was studied by Contact Angle measurement.
27 Moisture Buffering experiments were conducted by exposing the surfaces to cyclic changes in
28 relative humidity. It was found that coating with wax particles resulted in more hydrophobic
29 surfaces with enhanced moisture buffering ability, whilst the rest of the coatings examined either
30 reduced moisture buffering drastically (wax film, lacquer) or were not sufficiently hydrophobic
31 (linseed oil).

32 **Keywords:** Moisture buffering, $MBV_{\text{practical}}$, natural coating, wood surface modification, wax
33 microparticles

34 **Introduction**

35 For centuries, wood has been used extensively by humankind. Renewability, strength, visual
36 appearance and good thermal insulation properties made it the material of choice for many

applications, including building construction and furniture manufacture. During recent decades, however, new materials such as concrete, steel and, most recently, synthetic polymers or plastics have gradually replaced wood and in some application areas timber has become almost entirely disregarded as a building material. On the other hand, recent environmental and sustainability concerns have driven industry to look for new substitutes for fossil-based materials and the demand for green and renewable materials, including wood, is increasing.

Another important property of wood is hygroscopicity or, in other words, the ability to attract, hold, and release water molecules [1]. Hygroscopicity is often considered to be a negative characteristic of wood, as the exposure of timber to wet conditions can create many end use problems for non-treated surfaces, when used both outdoors and indoors. Excess water causes dimensional instability in the material due to swelling and shrinkage of the cell wall and lumen. Additionally, wet conditions create a very favorable environment for the growth of various wood degrading biological organisms (e.g. diverse fungi, bacteria, and insects). Therefore, it is very important to understand wood-water interactions and, if necessary, alter them [2-6]. To avoid problems of wood degradation and to enhance durability and easy maintenance, wood is often hydrophobized. Surface hydrophobization methods include, but are not limited to, treatments with silicon containing compounds [7-9], the deposition of metal oxide nanoparticles [10, 11] and surface impregnation with various waxes, oils, polyelectrolytes and other compounds [12-15]. Many of these approaches have a negative environmental impact and cause damage to the ecosystem because of the possibility for biocidal chemicals to leak from the surface [16].

Nevertheless, recent findings suggest that the hygroscopicity of wood can also be rather beneficial. The ability of a wooden material to store and release moisture helps it to regulate the indoor climate naturally, and to decrease humidity variations. This phenomenon, known as moisture buffering, is

an efficient way of passively moderating the moisture level in a living space [17, 18]. The use of hygroscopic materials together with a well-controlled ventilation system may further reduce the energy consumed for heating and cooling and increase the overall energy-efficiency of a building [19, 20]. Maintaining certain levels of relative humidity (RH) will also increase the perceived air quality and influence the occupants' health and comfort [21-24]. Especially during hot periods, the dynamic moisture storage in hygroscopic materials reduces the moisture in the air, leading to increased comfort and consequently a reduced need for cooling, resulting in *indirect* savings. However, it has been shown that since the indoor air humidity is reduced, the indoor air enthalpy is also reduced and consequently less energy is needed for cooling, which leads to *direct* energy savings [19]. The storage of moisture inside a hygroscopic material such as wood also means thermal storage, which can lead to passive heating or cooling of the building during the adsorption and desorption of water, and increased thermal comfort [25, 26].

To maximize the effect of moisture buffering, the surface area of wooden materials in interiors should be increased. This could be achieved by introducing more wooden surfaces into a living space, e.g. wooden floors, walls, ceilings, furniture, etc. Timber used indoors is certainly less susceptible to UV- and biodegradation by living organisms compared to wood used outdoors, but certain modification might still be required to improve the material properties and increase the lifetime of the material.

In order to be able to utilize the moisture buffering capabilities of timber, the influence of surface coatings and modification techniques should be studied with special care. However, whilst comprehensive literature is readily available on the topic of wood modification [1, 27, 28], little information could be found on the influence of finishings or coatings on the moisture buffering performance of timber. Studies conducted on various building materials, including wood,

generally suggest that paints and coatings decrease the moisture buffering effectiveness of the treated surfaces [29-33].

The aim of this work was to develop modification techniques that preserve the natural ability of wood to buffer moisture vapor while increasing its resistance to liquid water. Special focus was placed on the sustainability of the approach and therefore only natural materials were used, making the treatment green and the final product nontoxic for humans and nature. The treatment developed is fundamentally different to conventional wood modification techniques. It does not form a continuous film on the surface as many commercial paints and lacquers do and therefore does not limit moisture buffering. Instead, the method is based on the effect of hydrophobic wax microparticles that form a discontinuous surface layer, which repels water, slowing down penetration, but allowing moisture vapor permeation. The performance of wooden surfaces coated with wax particles was compared with that of a solid wax film and commercially available wood treatments (lacquer and linseed oil) in terms of water repellency and moisture buffering efficiency. Carnauba wax was chosen for the production of the wax particles because it is the hardest of the natural waxes and may, therefore, produce a durable coating. It is a natural wax produced by the leaves of Carnauba palm and has a melting point of 83-86°C [34]. This wax is also hypoallergenic, chemically inert and is not a food source for humans, and thus does not raise ethical issues when used in wood modification.

2. Experimental

2.1. Materials

Kiln dried spruce boards were obtained from a sawmill in Finland. For comparative purposes, all experiments were carried out on both radially and tangentially cut surfaces.

2.1.1. Wax coatings

Refined carnauba wax was purchased from Sigma Aldrich in the form of pellets.

The wax particles used to form the discontinuous coating were produced by melting the wax and dispersing it in water using a Polytron homogenizer PT2000 (Kinematica AG). The wax particles that were obtained were mostly spherical in shape and had a size distribution ranging from hundreds of nanometers to tens of micrometers. In order to make the coating less visible on the surface, the dispersion was filtered using qualitative grade paper filter retaining particles bigger than 12-15 μm . After filtration, the suspension was stable and the concentration was around 3.5 g/L. In this article, final dispersion is further referred to as colloidal wax particles. The filtered dispersion was applied to the wooden surface, allowed to dry and then buffed with a cotton cloth to distribute the particles evenly.

A continuous carnauba wax film coating was obtained by dipping the wood sample into molten wax. A thicker film was achieved by increasing the number of immersions of the sample into the molten wax. The area density was used to estimate the thickness of the coating and averaged 138 g/m^2 for the thin wax coating and 380 g/m^2 for the thick coating.

2.1.2. Commercial coatings

Two coats of commercially available linseed oil (Pellavaöljy, Tikkurila paints Oy, Finland) and spray lacquer (PROF, Matta spraylakka, Rautakesko Ltd., Finland) were applied to the wooden samples, following the manufacturers instruction.

2.2. Moisture buffering

The moisture buffering experiments were performed in accordance with the NORDTEST method [17]. Experimental setup is schematically represented in Figure 1. In order to evaluate the effectiveness of moisture buffering, the Practical Moisture Buffer Value ($MBV_{\text{practical}}$ ($\text{kg}/(\text{m}^2 \text{ \%RH})$)) was calculated. $MBV_{\text{practical}}$ defines the amount of moisture transported in to or out of a material per unit of open surface area, during a specified period of time, when the material is exposed to cyclic variations in relative humidity. In order to determine $MBV_{\text{practical}}$, the weight gain during absorption and the weight loss during drying were calculated, then averaged and normalized per open surface area and ΔRH . Average of weight gain and weight loss is taken for each cycle, and consequently $MBV_{\text{practical}}$ is calculated as the average of three cycles [14]. Further in this work, $MBV_{\text{practical}}$ will be referred to simply as MBV.

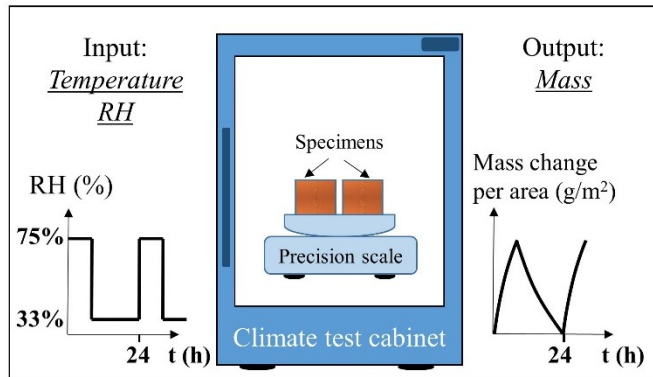


Figure 1. Schematic representation of experimental setup for the determination of MBV

Wood samples, around 2 cm thick, were sealed with aluminum adhesive tape on all but one side, leaving 4 cm^2 of surface available for moisture buffering. For each treatment, two parallel samples were tested. Before the measurements, all samples were pre-conditioned at $23 \pm 5^\circ\text{C}$ and $50 \pm 5\%$ relative humidity in a conditioned room until the weight of each specimen was stable. Cyclic changes in RH for the tests were obtained using a climate test cabinet (Rumed 4201, Rubarth,

Apparate GmbH, Germany) by ultrasonic humidification. The balance used for mass monitoring was a New Classic MS 204S with a precision of 0.01 mg (Mettler Toledo, Switzerland). Deviations from the standard NORDTEST method were the air velocity, which varied between 0.10 and 0.06 m/s instead of 0.10 ± 0.05 m/s, and the size of the samples.

During the moisture buffering measurements, the samples were exposed to cycles of high (75% for 8 hours) and low (33% for 16 hours) levels of RH. The duration of one full cycle was 24 hours. The mass of the specimens was recorded at the end of both RH periods and at four times during the period when the specimen was exposed to a RH of 75%.

2.3. Water Contact angle (CA)

The contact angle of water on unmodified and coated wooden surfaces was determined using a Contact Angle Meter CAM 200 (KSV Instruments Ltd., Helsinki, Finland) contact angle instrument with a computer based control system with video capture. The static sessile drop method was employed in the measurements and the CA was determined in at least three positions on each sample. The tests were performed with millipore water at room temperature, and a droplet volume of 6.7 μ L was used. The full Young-Laplace equation was used to determine the contact angle from the shape of the sessile drop.

2.4. Imaging and topography characterization

Scanning Electron Microscopy (SEM) was performed with a JEOL JSM-7500FA (JEOL Ltd., Tokyo, Japan) analytical Field Emission Scanning Electron Microscope using 2-5kV acceleration voltage. In order to avoid charging and to enhance the signal from the sample, the wooden specimens were sputter coated with gold before the imaging.

Topography maps were obtained with a 3D Optical Microscope ContourGT-K 3D (Bruker Corporation, Massachusetts, USA). Vision 64 onboard software was then employed to analyze these data and to calculate the area roughness parameter (S_q).

Atomic Force Microscopy (AFM) imaging in air was used to observe the distribution and size of the wax particles. A Nanoscope V MultiMode scanning probe microscope (Bruker Corporation, Massachusetts, USA) was used in tapping mode. Silicon cantilevers (NSC15/AIBS, MicroMasch, Tallinn, Estonia) with driving frequencies around 300-360 kHz were used for imaging. According to the manufacturer, the radius of the tip was less than 10 nm. The surface of each sample was imaged in at least three different places.

3. Results and Discussion

In order to preserve the moisture buffering effect of wood but still improve resistance to liquid water, a method based on surface coating using colloidal wax particles was investigated.

3.1. Moisture buffering performance

During the moisture buffering experiments, the wooden samples were subjected to cyclic changes in RH, imitating daily changes in the humidity of a living space. Moisture uptake and release are represented as mass per unit area for the tangential and radial surfaces in Figure 2.

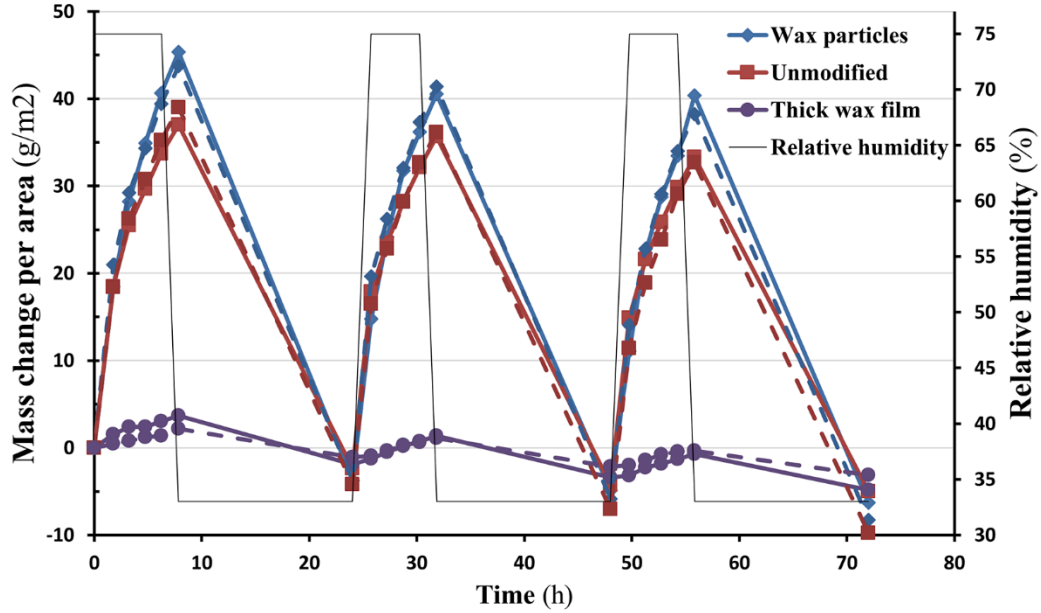


Figure 2. Moisture release and uptake cycles for tangential (solid line) and radial (dashed line) surfaces. The gray line shows the relative humidity in the chamber. The lines on the graph connect measuring points and do not correctly illustrate the rate of change if points are missing.

For both unmodified wood and wood treated with wax particles there was a clear increase in mass upon an increase in RH, and when the RH decreased, moisture was released from the wood sample. The samples with a continuous wax film did not react much to changes in RH. Declining tendency of the mass in Figure 2 can possibly be attributed to the fact that moisture buffering experiments were simulating daily changes in relative humidity and the “dry” period was much longer than the “wet” period (75% RH for 8 hours and 33% for 16 hours). Additionally, desorption of moisture is faster than adsorption when the relative humidity is high [35], and this could also have played a role.

The MBV values for the radially and tangentially cut surfaces with various treatments are shown in Figure 3. The unmodified radial and tangential surfaces displayed very similar sorption behavior

193 when subjected to RH changes, corresponding to MBV values of 0.98 and 0.91 kg/(m² %RH).
194 MBV of unmodified spruce that were found in literature appear to be somewhat higher, ranging
195 1.15-1.36 [17, 32]. However, the authors do not specify the direction of grain in the board, which
196 could greatly influence the obtained MBV values. As expected, all film-forming treatments
197 decreased the reference MBV of wood, possibly due to sealing of the pores and the isolation of
198 hydrophilic groups. Coating with wax particles, on the other hand preserved the buffering ability,
199 and even slightly enhanced it, increasing the MBV up to 1.08 on the radial and 1.09 on the
200 tangential surfaces. Therefore, according to the NORDTEST [17] classification, wood treated with
201 wax particles exhibits a good level of moisture buffering ($1 < \text{MBV} < 2$), whilst unmodified
202 specimen show only a moderate level of moisture buffering ($0.5 < \text{MBV} < 1$). Solid wax films on the
203 other hand reduce the buffering ability to limited ($0.2 < \text{MBV} < 0.5$) or, even, negligible levels
204 ($\text{MBV} < 0.2$) depending on the amount of wax applied. From this data, it can be concluded that
205 using particles is more advantageous compared to using a continuous wax film in terms of
206 preserving the natural moisture buffering efficiency of wood. Commercially available treatments
207 were also found to decrease moisture transport, resulting in a moderate level of moisture buffering
208 for the linseed oil coating and a limited level for the lacquer coating.

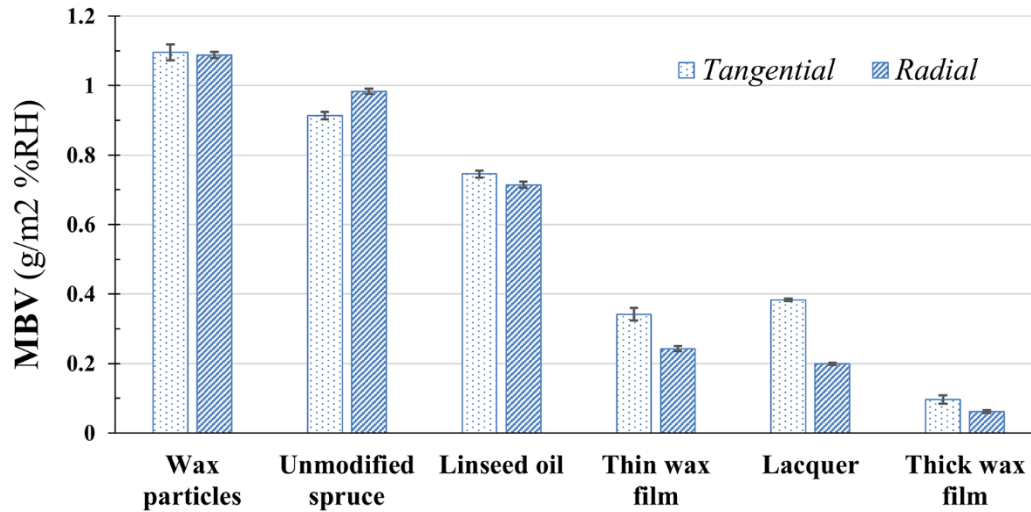


Figure 3. Practical Moisture Buffer Values for wooden surfaces with different treatments (radial and tangential surface).

Interestingly, an increase in the MBV of samples coated with wax particles most probably cannot be attributed to the chemical nature of the wax as it is hydrophobic and does not absorb water. However, as will be shown later (Table 1) the wax particles increase the roughness of the surface and this may have a positive effect on the moisture buffering.

3.2. Hydrophobicity of the treated wood

In this work the possibility of developing a coating for wood that repels liquid, slowing down the penetration of water, but allowing moisture vapor permeation and moisture buffering has been explored. The hydrophobicity of the surface has been studied by means of water CA measurements and results are presented in Figure 4. Standard deviation of CA for wax film and lacquer coating never exceed the value of around 3° and thus is not shown for the simplicity of the graph.

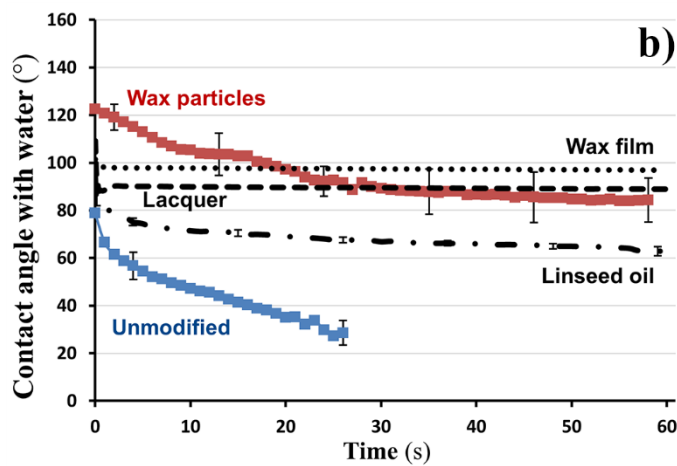
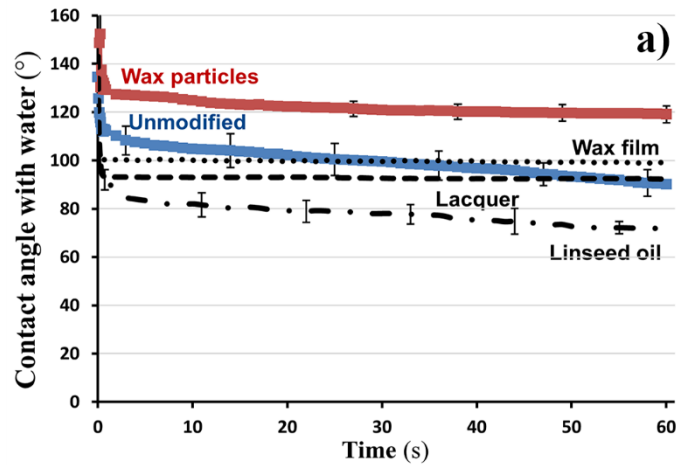


Figure 4. Water CA on unmodified and coated tangential (a) and radial (b) wooden surfaces. Vertical bars represent standard deviation.

Film-forming coatings like the layer of solid wax and the lacquer were found to completely prevent water penetration through surface, leaving the water droplet to rest on the surface and slowly evaporate. This can be seen in Figure 4 as a constant value of CA around 90-100° for the wax film and lacquer regardless of the type of surface (i.e. radial or tangential). These findings are in good agreement with the low MBV of the corresponding coated surfaces, both indicating the impermeability of the coated surface to water.

It is known that surface roughness and surface energy are the main factors influencing wettability [36]. Hierarchical surface roughness paired with a low surface energy of the material might result in a surface with exceptional hydrophobicity [37, 38]. In this work, the combination of naturally rough timber with colloidal wax particles having low surface energy were found to influence the overall hydrophobicity of a wooden surface, making it more resistant to liquid water, and increasing CA after the first minute from 0 to 80° for a radially cut surface and from 90 to 120° for a tangential cut. Interestingly, the tangential surface treated with wax particles had a higher CA, at 120°, than the surface with a continuous wax film, on which the CA was 100°. This increase in CA values can possibly be attributed to increased complexity of surface roughness of timber created by particles of different sizes. Initially rough surfaces with the addition of particles might result in the formation of air pockets with entrapped air, preventing the water droplet penetrating through the surface. However, perhaps due to high heterogeneity and porous nature of the wooden material, coating with wax particles does not completely prevent water from spreading and permeating through the treated surface. As a result, a slow decrease of CA value over time is observed.

Treatment with natural linseed oil was found to preserve a moderate level of moisture buffering as was mentioned earlier. However, the coating was also found to be the least efficient against liquid water, making the tangentially cut surface appear even more hydrophilic than the unmodified specimen. Thus, linseed oil has negligible use when enhancing the resistance of timber to liquid water.

3.3. Morphology of coated wooden surfaces

In Figure 5, SEM images of the timber coated with wax particles (a) and with a continuous wax film (b) are shown. The particles (Fig. 5a) were found to be mostly spherical in shape and to be distributed rather evenly, covering the wooden surface. Smaller spheres (less than 1 μm in diameter) seemed to form aggregates of several particles. The size distribution is illustrated in the AFM image in Figure 6. A solid layer of wax, on the other hand, appears to be smooth and continuous (Fig. 5b), completely covering the natural features of the wood surface. The small structures seen on the surface were probably formed during air-drying of the film.

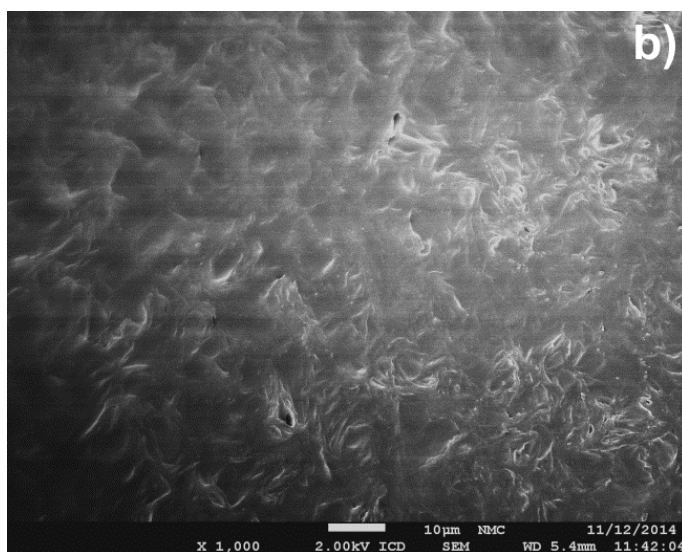
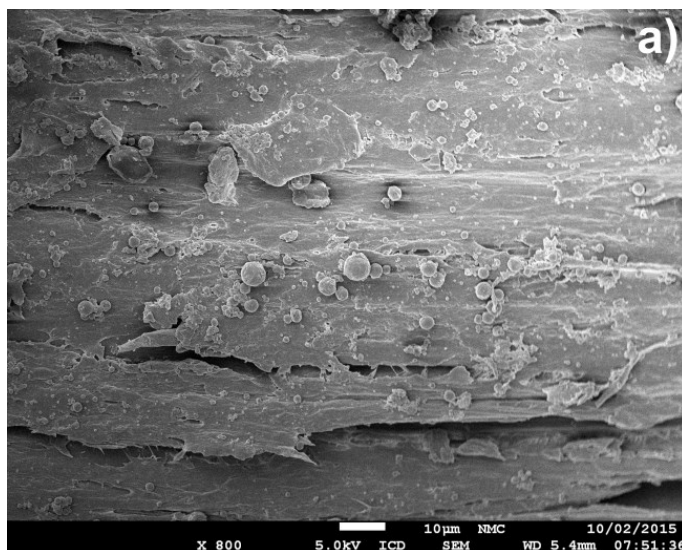


Figure 5. SEM images of wood coated with wax particles (a) and with solid layer of wax (b).

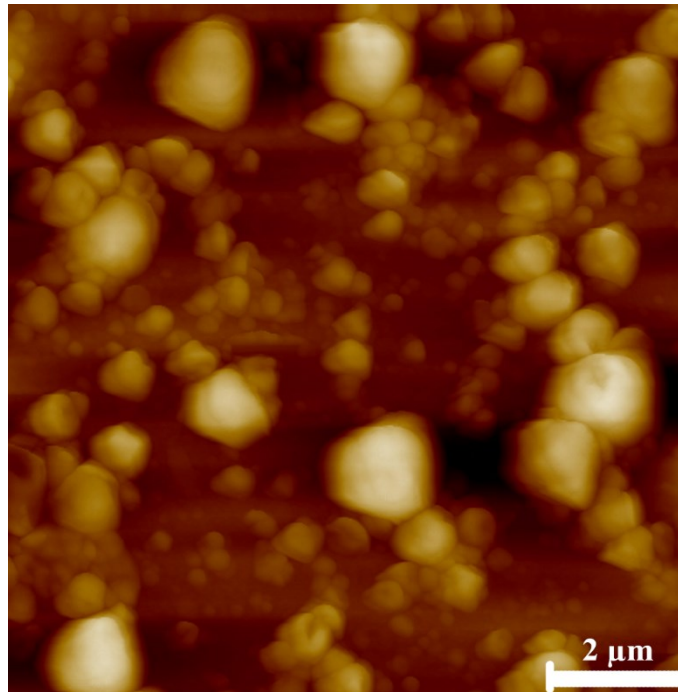


Figure 6. AFM height image of wax dispersion filtrated with 1μm pore size filter and dried on mica substrate. The image was scanned in air.

To gain further understanding of the roughness of the final coated surfaces, topography maps were acquired with a 3D optical microscope and the area roughness parameters (S_q) were calculated for an area of 0.3 mm² on each sample. As can be seen from Table 1, a solid layer of wax decreases the initial roughness of the surface drastically, whilst wax particles actually make surface slightly rougher. This data correlates well with the previous findings on CA values, explaining the very high initial CA of the coating with wax particles (150°) and the lower CA of the wax film (100°), as increased roughness is known to be required for outstanding surface hydrophobicity [3]. Furthermore, the increased roughness could also possibly explain the high MBV value of the wood samples treated with colloidal wax particles.

Table 1. Area roughness parameters for unmodified and coated timber.

	Sq (μm)
Unmodified wood	14.9
Continuous wax film	4.4
Wax particles	17.6

The designed coatings will allow an optimized use of wood in living spaces due to improved resistance to liquid water and enhanced moisture buffering ability. The increased hydrophobicity of a wood surface may allow it to be used in wet spaces and possibly make it easier to clean and maintain.

Conclusion

The hydrophobization of a wooden surface was achieved through coating with wax microparticles dispersed in water. This treatment increased the surface roughness of wood, whilst forming a discontinuous surface layer, repelling water and slowing down its penetration. The moisture-vapor sorption properties of wood, however, stayed intact and therefore the moisture buffering ability of the material was retained and even slightly enhanced.

Film forming coatings, such as melted wax and lacquer were found to improve the hydrophobicity of the wood surface, but to drastically decrease the moisture buffering ability, reducing it to a limited or even negligible level. Coating with linseed oil only partially reduced the moisture buffering performance, however, it left the tangentially cut surface more hydrophilic than the unmodified one.

In this work, an example was shown using wax microparticles. To our knowledge, this is the first demonstration of a surface treatment that introduces hydrophobicity but retains the moisture buffering effect of wood. We suggest that the general idea of using a non-continuous layer of particles to modify wood surfaces could be used in various applications. For example, introducing easy maintenance, water resistance, fire retardant, antimicrobial/antifungal properties or even as an alternative to painting to introduce coloring. The benefits of this approach is the retained moisture buffering capacity and possibility for passive climate control.

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